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SOLID LUBRICANT COATINGS CURABLE AT 225 F-300 F

George Murphy, Jr.

Army Weapons Command
Rock Island, Illinois

July 1972

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TECHNICAL REPORT

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ABSTRACT

Experimental solid-film lubricant coatings based on urea-formaldehyde, epoxy-polyamide, epoxy-silane, alkyd-urea, melamine-acrylic, and epoxy-urea resins were formulated by personnel of the Research Directorate, Weapons Laboratory at Rock Island. None of these coatings when cured at temperatures of 225-300°F had antiwear or corrosion preventing properties comparable to the fully cured MIL-L-46010A type of solid lubricant coating. Of the experimental formulations tested, those based on the urea-formaldehyde and epoxy-silane resins gave the best results. However, with the addition of a curing agent, boron trifluoride monoethylamine complex, to a qualified MIL-L-46010A product, the product could be cured at 275°F, and all test requirements of Military Specification MIL-L-46010A could be satisfied.

FOREWORD

This investigation was carried out under DA Project 1G062105A107, AMS Code 502E.11.80100.01, "Lubricants, Friction and Wear." The unit under which the study was made is entitled "Wear and Corrosion Preventive Resin-Bonded Solid Film Lubricant Curable at 225°F."

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13. ABSTRACT

Experimental solid-film lubricant coatings based on urea-formaldehyde, epoxy-polyamide, epoxy-silane, alkyd-urea, melamine-acrylic, and epoxy-urea resins were formulated by personnel of the Research Directorate, Weapons Laboratory at Rock Island. None of these coatings when cured at temperatures of 225-300°F had antiwear or corrosion preventing properties comparable to the fully cured MIL-L-46010A type of solid lubricant coating. Of the experimental formulations tested, those based on the urea-formaldehyde and epoxy-silane resins gave the best results. However, with the addition of a curing agent, boron trifluoride monoethylamine complex, to a qualified MIL-L-46010A product, the product could be cured at 275°F, and all test requirements of Military Specification MIL-L-46010A could be satisfied. (U)

(Murphy, George Jr.)

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14 KEY WORDS	LINK A		LINK B		LINK C	
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1. Lubrication						
2. Corrosion Protection						
3. Solid Film Lubricants						
4. Resins						
5. Cure Temperature						
6. Curing Agents						

OBJECTIVE

The objective of this study was to develop a solid-film lubricant coating with properties equivalent to those of the MIL-L-46010A type of solid lubricant coating, but curable at 225°F to 300°F.

BACKGROUND

Solid-film lubricants are currently used extensively on weapons such as the M16A1 rifle. From the point of view of wear prevention, corrosion protection, adhesion, and fluid resistance, the MIL-L-46010A[1] type of solid-film lubricant coating is adequate for many weapons applications. However, this coating has the disadvantage that its recommended cure temperature of 400°F causes deterioration of some of the alloys used in weapons such as the M16A1 rifle. For example, at temperatures above 300°F, the yield strength, tensile strength, elongation and impact resistance of 7075 and 6061 aluminum alloys are adversely affected.[2,3,4]

Because of this detrimental temperature effect, the fully cured MIL-L-46010A coating cannot be used on these aluminum alloy parts. The solid-film lubricant coatings currently used on aluminum parts are either commercially available proprietary materials which are cured at 250°F - 300°F or the MIL-L-46010A coating cured at 300°F. None of these are as effective as the fully cured MIL-L-46010A coating.[5] Therefore, a need exists for a solid-film lubricant curable at 225°F-300°F that has the properties of the fully cured MIL-L-46010A type coating.

The determining factor at which temperature a bonded solid-film lubricant coating can be cured is the temperature at which its resin binder is fully cured. A survey of the literature indicates which types of resins are fully cured at a temperature of 225°F-300°F. The literature shows that the following types of organic resin systems are feasible: (1) urea-formaldehyde, (2) epoxy-polyamide, (3) epoxy-silane, (4) alkyd-urea, (5) melamine-acrylic, and (6) epoxy-urea.

Payne[6] states that the urea-formaldehyde-based finishes are normally baked at 250°F-300°F. However, acid catalyzed urea-formaldehyde-based finishes have been cured at 140°F-160°F. Epoxy-polyamide is basically an air-cure system in which a reaction between the amino group in the

polyamide and the epoxy resin forms a cross-linked copolymer. The epoxy-silane resin is a commercial experimental material curable at 250°F which is reported to give good corrosion protection. This resin was developed as a primer coating. Alkyd-urea resins are cured in the same temperature range as the urea-formaldehyde resins (250°F-300°F) and have similar properties; however, they are not as brittle as the urea-formaldehyde coatings. The melamine-acrylic resins are cured at 250°F to give a hard, resistant film. The urea-formaldehyde resins, which are amino in characteristics can also cross-link with epoxy resins when cured at 300°F-325°F.

APPROACH

Formulation Work

Based on information on properties of resin systems gained from the literature, a number of formulations were made and were compared with existing solid-film lubricants. The initial formulations that were considered best were varied in composition and cure conditions to find the optimum combination.

Modification of Existing Formulation

A curing agent, boron trifluoride monoethylamine complex, was added to an existing MIL-L-46010A formulation to lower the curing temperature of the solid-film lubricant. The modified coating was tested against the specification requirement of Military Specification MIL-L-46010A.

PROCEDURE

Formulation Work

Formulations that contained approximately 27 to 30 per cent by weight resin and 70 to 73 per cent by weight pigment system were prepared. Compositions of these formulations are given in Table I. These formulations were tested for storage stability by periodic examination for evidence of gelation or livering. The lubricant formulations were also subjected to the wear life test of MIL-L-46010A specification. In this test, the coating is applied to zinc phosphatized test specimens, the coated specimens are cured for 2 hours at 225°F, and then the endurance life of the coatings is determined on the Falex Wear Tester in accordance with Method 3807.1 of Federal Test Method Standard 791B.[7]

TABLE I

INITIAL SOLID LUBRICANT FORMULATIONS

Component	Formulation (% by weight) MA-1	FA-1	FA-2	EU-1	EP-1	EP-2	EP-3	U-1	ES-1
Resin (Type)									
Acrysol WS-24 (Acrylic)	24.0								
Uformite MM83 (Melamine)	5.0								
Duraplex A-29 (Alkyd)		21.0	21.0						
Uformite F200E (Urea)		9.0	9.0	9.0					
Epon 1007 (Epoxy)				21.0					
EKR 2002 (Epoxy)					18.0	16.0	18.0		
Versamid 115 (Polyamide)					9.0	9.0	5.0		
Plaskon 3300 (Urea)								27.0	
XZ-8-5059 (Epoxy-Silane)									30.0
Percent Resin	29.0	30.0	30.0	30.0	27.0	25.0	23.0	27.0	30.0
Pigment									
Molybdenum Disulfide	38.0	37.0	37.0	37.0	39.0	40.0	41.0	38.0	37.0
Antimony Trioxide	24.0	24.0	24.0	24.0	25.0	26.0	26.0	25.0	24.0
Dibasic Lead Phosphite	9.0	9.0	8.5	8.7	9.0	9.0	10.0	9.0	9.0
Percent Pigment	71.0	70.0	69.5	69.7	73.0	75.0	77.0	72.0	70.0
Catalyst									
Phosphoric Acid								1.0	
Paratoluenesulphonic Acid			0.5	0.3					

When the formulations having the best storage stability and wear life were determined, variations of these materials were made to find the optimum combination of resin, pigment, catalyst, and cure conditions that would give the longest wear life.

Modification of Existing Formulation

A concentration of 0.6 per cent by weight boron trifluoride monoethylamine complex was added to a qualified MIL-L-46010A product. The formulation was applied to test specimens and cured under several conditions of time and temperature; it was then tested for solvent resistance to dioxane and wear life according to the procedures of specification MIL-L-46010A. When the optimum cure conditions were determined, the BFMEA modified MIL-L-46010A coating was subjected to all MIL-L-46010A specification tests.

RESULTS AND DISCUSSION

Formulation Work

The results of the Falex wear tests on the initial experimental formulations and some commercial formulations after a 2-hour cure at 225°F are given in Table II. The data show that only one formulation, ES-1, was comparable in wear life to the commercial formulations identified as 626, 99A, and RIAPD 703. However, all formulations were comparable to, or better than, the MIL-L-46010A coating in wear life when cured at 225°F instead of at the normal 400°F cure. The data also show that the wear life of none of the experimental lubricants was comparable to the 500 minute wear life obtainable for the fully cured MIL-L-46010A coating.

In addition to wear life, the gelation time of the experimental formulations was also determined The number of months required for gelation of the formulation to occur is given in Table III. When gelation occurs, the formulation is no longer useful. If this gelation occurs in less than 6 months, the formulation is considered to have poor storage stability. The data in Table III show that, for the experimental formulations, only those based on the urea-formaldehyde and epoxy-silane binder had sufficient storage stability to be considered for further formulation work.

TABLE II

SOLID LUBRICANT TEST DATA,
INITIAL FORMULATIONS (CURED 2 HOURS AT 225°F)

Solid Lubricant Formulation	Falex Wear Life (Minutes)
MA-1	17
FA-1	3
FA-2	21
EP-1	13
EP-2	12
EP-3	22
EU-1	91
U-1	19
ES-1	117
MIL-L-46010	15
MIL-L-46147	160
626	92
99A	148

TABLE III

GELATION TIME OF INITIAL FORMULATIONS

Solid Lubricant Formulation	Gelation Time (Months)
MA-1	2.0
FA-1	4.0
FA-2	4.0
EP-1	0.5
EP-2	0.5
EP-3	0.5
EU-1	3.0
U-1	14.0^{+}
ES-1	14.0^{+}
MIL-L-46010	6.0^{+}
MIL-L-46147	6.0^{+}
626	6.0^{+}
99A	6.0^{+}

The composition of additional formulations used in the optimization study is given in Table IV, and the wear life of these formulations when tested under various cure conditions is given in Table V.

Of the epoxy-silane formulations, ES-2 is optimum. This formulation, however, was only slightly better than ES-1, which is essentially the same as ES-2 except that it contained no catalyst. Increasing the cure temperature over the range 225°F-300°F had no effect on the wear life of the epoxy-silane solid lubricant coatings.

For the urea-formaldehyde type of formulations, U-2 is optimum because wear life decreased when the amount of resin was either greater or less than 32 per cent. The data also show that, for the urea-formaldehyde type of coatings, wear life is dependent upon the cure temperature.

Corrosion tests of formulations ES-2 and U-2 showed that both of these formulations protected the zinc-phosphated steel panels from rust for about 70 hours in the salt fog test when these panels were cured for 2 hours at 275°F. Although these formulations do not have properties equivalent to those of the fully cured MIL-L-46010A coating (100 hours minimum protection in the salt fog test), the results are encouraging and show that development of a low cure-temperature coating equivalent to standard MIL-L-46010A may be possible.

Modification of Existing Formulation

The MIL-L-46010A formulation with 0.6 per cent catalyst, boron trifluoride monoethylamine,added was found to be curable to the solvent resistant state in 45 minutes at 275°F. Data on wear life of the modified coating under various cure conditions are shown in Table VI.

These data show that, although the coating is solvent-resistant after a 45-minute cure at 275°F, a 120-minute cure at this temperature is necessary to obtain a Falex wear life by which the MIL-L-46010A wear life requirement is satisfied. Since the 120-minute cure at 275°F was the lowest cure conditions at which the modified MIL-L-46010A coating would meet the Falex wear life requirement, these conditions were used to fully evaluate the modified coating. The results of this evaluation are given in Table VII. These results

TABLE IV

OPTIMIZED SOLID FILM LUBRICANT FORMULATIONS

Component	Formulation (% by weight)						
	ES-1	ES-2	ES-3	U-1	U-2	U-3	U-4
Resin (Type)							
XZ-8-5059 (Epoxy-Silane)	30.0	30.0	35.0				
Plaskon 3300 (Urea)				27.0	32.0	35.0	37.0
Percent Resin	30.0	30.0	35.0	27.0	32.0	35.0	37.0
Pigment							
Molybdenum Disulphide	37.0	37.0	35.0	38.0	36.0	34.0	33.0
Antimony Trioxide	24.0	24.0	22.0	25.0	23.0	22.0	21.0
Dibasic Lead Phosphite	9.0	8.5	8.0	9.0	8.0	8.0	8.0
Percent Pigment	70.0	69.5	65.0	72.0	67.0	64.0	62.0
Catalyst							
Phosphoric Acid		0.5		1.0	1.0	1.0	1.0

TABLE V

FALEX WEAR LIFE (MINUTES), OPTIMIZED FORMULATIONS

Formulations / Cure Conditions	ES-1	ES-2	ES-3	U-1	U-2	U-3	U-4
2 hours at 225°F	117	133	88	19			
2 hours at 275°F	125			267	334	90	55
1 hour at 300°F	120			400			

TABLE VI

EFFECT OF CURE CONDITIONS ON FALEX WEAR LIFE
OF MODIFIED MIL-L-46010A COATING

Cure Time (Minutes)	Cure Temperature (°F)	Falex Wear Life (Minutes)
60	250	22
120	250	15
45	275	15
60	275	55
120	275	800
120	300	738

TABLE VII

SPECIFICATION TEST RESULTS FOR BFMEA MODIFIED MIL-L-46010A SOLID-FILM LUBRICANT

Test	Test Requirement	Test Results
Solids Content	Minimum 40%	40.6
Film Thickness	.0002-.0005 inches	.0005 inches
Wear Life	Minimum 450 minutes	800
Load-Carrying Capacity	Minimum 2000 pounds	3250
Corrosion Protection	Minimum 100 hours	500+
Film Adhesion	Coating not removed by tape	Pass
Fluid Resistance		
Trichloroethylene	Pass film adhesion	Pass
Carbon Removing Compound	Pass film adhesion	Pass
Standard Hydrocarbon Test Fluid	Pass film adhesion	Pass
Rifle Bore Cleaner	Pass film adhesion	Pass
Aviation Gasoline	Pass film adhesion	Pass
Petroleum Base Hydraulic Fluid	Pass film adhesion	Pass
Jet Fuel	Pass film adhesion	Pass
Aircraft Lubricating Oil	Pass film adhesion	Pass
Nonpetroleum Base Hydraulic Fluid	Pass film adhesion	Pass
Silicate Ester Type Lubricating Oil	Pass film adhesion	Pass
Diester Type Semifluid Lubricating Oil	Pass film adhesion	Pass
Silicone Damping Fluid	Pass film adhesion	Pass
Synthetic Aircraft Turbine Lubricating Oil	Pass film adhesion	Pass
Thermal Stability	Pass film adhesion	Pass
Graphite and Powdered Metals	None	None
Storage Stability (6 months)		
Wear Life	Minimum 450 minutes	608
Corrosion Protection	Minimum 100 hours	300
Workmanship	Completely dispersed by moderate shaking or stirring	Pass

show that the modified coating meets all the specification requirements, including the six months storage stability test. The Falex wear test was also run on the modified coating after 8 months, and a wear life of 20 minutes was obtained. This shows that the catalyst was no longer effective since 20 minutes is the wear life of the unmodified MIL-L-46010A coating when cured for 2 hours at 275°F.

CONCLUSIONS

1. Of the low temperature-cured experimental formulations tested, only those based on urea-formaldehyde or epoxy-silane binder have antiwear, corrosion preventive, and storage stability properties that approach those of the fully cured MIL-L-46010A coating. However none of the materials tested matched the performance of the fully cured MIL-L-46010A coating.

2. The addition of boron trifluoride monoethylamine to the MIL-L-46010A solid-lubricant formulation decreased the cure temperature from 400°F to 275°F without an unfavorable effect on the properties of the coating.

RECOMMENDATION

Since the work with the boron fluoride curing agent was limited to one qualified MIL-L-46010A lubricant, further investigation should be carried out to determine its effect on other qualified MIL-L-46010A lubricants before a final recommendation for the use of the curing agent can be made.

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